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CHEMISTRY AND TECHNOLOGY OF FUELS AND LUBRICANTS (SELECTED ARTICLES)

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CHEMISTRY AND TECHNOLOGY OF FUELS AND LUBRICANTS (SELECTED ARTICLES)

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SYNTHESIS AND PROPERITES OF ARYLSTEARIC ESTERS

M.G. Rudenko, Yu. P. Sobolev, M. S. Yatsenko, L. B. Starikova

The first of the series of aryl stearic acids to be synthesized was phenylstearic acid by Marcusson [1] in 1920 by condensations of benzene with oleic acid in the presence of AlCla.

There followed a number of papers on the improvement of the method of preparing phenylstearic acid proper as well as other acids in this series.

Arylstearic acids with the following aryl groups have been synthesized: phenyl, tolyl, naphthyl, phenoxyphenyl and others.

For these only three esters have been reported: ethyl tolylstearate, methyl phenoxyphenylstearate, and methyl phenylstearate (not characterized) [2 through 8].

Among the number of patents that have been applied for is one for the application of arylstearic acids and their salts as additives to mineral oils [9].

The present paper is devoted to the synthesis and study of the properties of esters of arylstearic acids in order to explore the possibility of using them as synthetic lubricating oils.

We prepared phenyl, o-xylyl, and p-xylylstearic acids by condensation of commercial oleic acid with benzene, o-xylene, and p-xylene in the presence of AlCl₃. The aromatic component in the reaction was taken in excess to oleic acid (5 to 1), while AlCl₃ and oleic acid were taken in equimolar amounts. The reaction was carried out at 80° for 5 to 6 hr. We purified the prepared arylstearic acids by distillation. In the arylation with naphthalene and diphenyl ether, trichlorbenzene was used as a solvent. Phenoxyphenylstearic acid, along with the above mentioned acids, was purified by distillation, but naphthylstearic acid was converted to methyl naphthylstearate immediately after being prepared and the stearate was later subjected to distillation. Of the acids prepared, phenoxyphenylstearic and o-xylylstearic had constants which differed from those given in the literature [6, 8].

	o-xylylstearic acid	phenoxyphenyl- stearic acid
boiling point at 0.015 mm, oc	201-202	245—251
	1,4975 (1,4936)	1,5198 (1,5263)
d 4	0,9388 (0,9336)	1,0032 (0,9964)
MR _D found	120,86 (121,1)	137,129 (138,09)
round calculated	120,2 (120,2)	136,835 (136,72)

(Data from the literature are given in parentheses)
The procedure followed enabled us to obtain higher yields for
some arylstearic acids than those stated in the literature.

The yields of the arylstearic acids are listed below (%).

	Given in the literature	Obtained
p-xylylstearic	67 [8]	65
Naphthylstearic	17.5 [8]	32
Phenoxyphenylstearic	29 [6]	41
Phenylstearic	38 [6]	39

The commercial oleic acid used in the reaction contained a certain amount of impurities (linoleic and stearic acids), and because of this, upon fractional distillation of the reaction mixture stearic acid or its ester distilled first and only then the ester of the corresponding arylstearic acid or the acid itself. A large pot residue always remained. It was apparently a misture of polysubstituted aromatic compounds with esters of polymers of linoleic acid, which appeared to be the reason for the low yields of arylstearic acids. Only in the case where xylol, a compound with two substituents in the ring, was used was a 65% yield of xylylstearic acid obtained.

The properites of the residue from methyl phenoxyphenylstearate were: solidification point = -20°; $\nu_{100} = 35.70$; $\nu_{50} = 279$. The properties of the residue from methyl naphthylstearate were: solidification point = -5; $\nu_{100} = 55.0$; $\nu_{50} = 522$.

We prepared the methyl, benzyl, and 2-ethylhexyl esters of the phenylstearic, o-xylylstearic, n-xylylstearic, naphthylstearic, and phenoxyphenylstearic acids which we had prepared and examined their properties (Table 1).

Almost all of the esters had very low solidification points ranging from -40° to -60° . The benzyl naphthyl- and benzyl phenoxyphenylstearates have a somewhat higher solidification point of -35° , whereas the methyl esters of the same acids solidify at -40° to -50° . The situation with the esters of phenylstearic acids is peculiar; the methyl ester solidifies at -26° , and the benzyl ester at -50° , although the viscosity of the latter ($\nu_{50} = 19.32$) is much higher than the viscosity of the methyl ester ($\nu_{50} = 11.38$).

At the same time, the esters of stearic acid are solids and have a high solidification point. As an example of this, the benzyl ester

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has a melting point of 45.8°, and the methyl ester, 38°.

TABLE 1
Properties of the esters of arylstearic acids

	boiling	ure 18	30	- 20	M	R _D	Solidifica-
Esters of arylstearic acids	point *C	ី ខែ	•4.	A D	Exper- lmental	Calcu- lated	point °C
Methyl phenylstearate	178—183	0,1	0,9220	1,4825	115.93	115,70	26
2-ethylhexyl phenylstear-	220—225	0,01	0,9038	1,4780	148,07	148,03	be}ow 60
Benzyl phenylstearate	224—229	0,015	0,9587	1,5075	140,140	139,810	-50
Methyl p-xylylstearate	178—180	0,015	0,9259	1,4880	125,29	124,94	-50
Benzyl p-xylylstearate	230—237	0,02	0,9616	1,5105	148,89	149,05	-45
Methyl o-xylylstearate	186188	0,015	0,9253	0,4886	125.41	124,94	-52
Benzyl o-xylylstearate	234—237	0,015	0,9597	1,5195	149,31	149,04	-50
Methyl naphthylstearate	220-224	0,02	0,9556	1,5220	135,53	131,04	-40
Benzyl naphthylstearate	242-252	0,015	0,9928	1,5400	158,24	155,15	-38
Methyl phenoxyphenylstear- ate	199203	0,015	0.9525	1,5125	142,56	141.45	50
Benzyl phenoxyphenylstear-	258—260	0,02	1.0068	1,5307	166,71	165,56	-35

The hypothesis has been previously stated that upon the arylation of oleic acid a mixture of isomers is obtained since an addition can take place st positions 9 and 10.

Harmon and Marvel [4] showed for the case of phenylstearic acid that it is actually a mixture of nearly equal parts of 9- and 10-phenylstearic acids. By malonic ester synthesis they obtained 9-phenylstearic acid with melting point of 36.5° to 38° and 10-phenylstearic acid with a melting point of 40° to 41.5°. The phenylstearic acid which these authors obtained from oleic acid was a liquid with a solidification point of about 0°; that obtained by us has a solidification point of -7°.

Thus, the lower solidification point of the esters of arylstearic acids prepared by us, compared with the esters of the stearic acids,

can be explained by the fact that they are not discrete compounds, but mixtures of isomers, and by the lowering of the solidification point upon the introduction of the aryl group. For example, the solidification point of stearic acid is 69.4° and that of 9-phenylstearic acid, 36.5° to 38°.

The viscosity of the esters (Table 2) increases for a particular acid with the increase in the number of carbon atoms in the alcohol, and for different acids with an increase in the molecular weight of the arylating reagent.

The viscosity of the esters of phenoxyphenyl stearic acid is lower than that of the esters of the naphthylstearic acid though the phenoxyphenyl radical is heavier than the naphthyl. In all probability this discrepancy is due to the ether function of the phenoxyphenyl radical.

The thermal oxidation stability of the arylstearates obtained was examined at 300° and with air bubbling through at the rate of 5 ml per minute for 10 hrs. The results of the tests are given in Table 3. Methyl phenoxyphenylstearate and benzyl p-xylylstearate gave better results by all indications in preventing corrosion of EI-347 steel. The test of thermal oxidation stability at 300° was conducted without the use of anti-oxidation and anti-corrosion additives.

In the oxidation of benzyl p-xylylstearate with the addition of .5% AzNII-10 there was observed a decrease in volatility from 16.5% to 3.6% and a decrease in corrosion of EI-347 steel from 162.4 g/m² to 0.0.

This data indicates that when matched with corresponding additives benzylp-xylylstearate and methyl phenoxyphenylstearate can be used

as lubricating materials or components of lubricating materials at 300°.

TABLE 2
Kinematic Viscosity of Esters of Arylstearic Acids

Esters of anylateanic acids	100*	50°	Temperatures below zero
Methyl phenylstearate 2-ethylhexyl phenylstearate Benzyl phenylstearate Methyl p-xylylstearate Benzyl p-xylylstearate Methyl o-xylylstearate Benzyl o-xylylstearate Methyl naphthylstearate Benzyl naphthylstearate Benzyl naphthylstearate Benzyl phenoxyphenylstearate Benzyl phenoxyphenylstearate	8,70 5,4 5,55 4,96 5,95 5,20 7,40 7,20 9,90 6,50 9,00	11.40 19.20 19.30 19.00 30.50 21.10 31.90 33.50 51.10 27.50 41,60	451 (-20° 15900 (-40° 5500 (-30° 11600 (-30° 13500 (-35° 24300 (-30° 14300 (-25° 66000 (-35°) 17600 (-30° 22500 (-25°)

TAHLE 3

Thermal Oxidation Stability of Esters of Arylstearic Acids at 300° for a Period of 10 hours

Estero :1	acid number, me		Precipi-	Volatil	Gerrosion of metals, g/m ²	
anylsteari; acids	initial	after oxida- tion	tate ≸	ity,	EI-347	AK-4
Methyl phenylstea ate	r 0,34	24 00	0,072	11,4	-104,7	0.0
Methyl o-xylylste	ay- 0,51	25.35	0,084	6.6	-27,6	-0.5
Methyl p-xylylste	0,45	26 .52	0,087	10,1	90,4	-o.:
ate Benzyl <u>p</u> -xylylste	a 0.93	4,95	0.017	16,5	-162,4	0,0
rate Methyl phenoxyphe oxyphenylstearate	n- 0,54	18 2	0,03	4,5	-3,1	Q,C
P. residue of methyl naphthylst	1.22	14.90	0,1	7,9	-51,4	0,0

Conclusions

1. A method for preparing phenyl, p-xylyl, o-xylyl, naphthyl,

and phenoxyphenylstearic acids has been developed.

- 2. Methyl, 2-ethylhexyl, and benzyl phenylstearate; methyl and benzyl p-xylyl- and o-xylylstearate; methyl and benzyl naphthyl-stearate; and benzyl phenoxyphenylstearate were prepared for the first time and their properties were examined.
- 3. All the esters of arylstearic acids prepared have low solidification points and a gently sloping viscosity - temperature curve.
- 4. The esters of arylstearic acids were tested for thermal oxidation stability at 300° for a period of ten hours. The best results were obtained for methyl phenoxyphenylstearate and benzyl p-xylylstearate. When matched with the corresponding anti-oxidation and anti-corrosion additives the esters mentioned can be used as lubricating materials or components of lubricating materials at a temperature of 300°.

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LESSENING THE CORROSIVE ACTION OF ANTI-ABRASIVE
ADDITIVES WITHOUT IMPAIRING THEIR EFFECTIVENESS

Yu. S. Zaslavskiy, G. I. Shor, R. N. Shneyerova, A. I. Kuznetsova, F. B. Lebedeva

The radiological studies performed have enabled us to formulate a clear statement of the essential difference in the requirements that must be met for the chemical compounds when matching anti-corrosive and anti-abrasive oil additives. This difference is summed up in the fact that anti-corrosive additives must have stable sulfur phosphorus bonds in their molecules, while the anti-abrasive additives should easily eliminate sulfur, phosphorus, or chlorine in order to form the appropriate sulfide, phosphide, or chloride of the metal at high contact temperatures [5-7]. Such an essential difference in the aforementioned properties of the additives explains the maximum anti-abrasive effectiveness which many authors have set for chemically active compounds that have a corrusive effect upon the metals which they protect from abrasion [8,9]. The mose effective anti-abbrasive additives can be obtained not only with the combination of chemical compounds in normal use, which would provide a satisfactory anti-abrasive effect with a minimum of corrosion [10-12], but also by matching of additives which combine the most effective anti-abrasive and anti-corrosive components. with such a composition of additives, it is apparently possible to eliminate an effect observed last time [13] in which the activity of the suti-abrasive additives is

delayed when they are used in friction-producing units of high-speed mechanisms. This effect is related to the fact that anti-abrasion additives are usually devised on the basis of a calculation of their chemical activity toward the metal of the surfaces of friction only after the additives have decomposed after being heated to certain high temperatures. It is assumed that the occurrence of such high temperatures on portions of the rapid-heating surfaces of friction will bring about the decomposition of some amount of additive near the heat-producing zone, while the main supply of additive remains unchanged in the volume of the oil. Apparently, in high speed mechanisms not enough time is provided at the surfaces of friction for decomposition of the additive and reaction of the decomposition products with the metal surfaces. This must be taken into account in the development of a composition for anti-abrasive additives.

For the study and selection of two-component additives under laboratory conditions we have devised radiological methods of determining the chemical activity of anti-abrasive additives in oil in the presence and absence of friction. The chemical activity of anti-abrasive additives, which determines their reactivity in connection with the metal which they protect from abrasion and seizing, and which thereby determines their anti-abrasive effectiveness[12, 14, 15], is appraised by determining the kinetics of the dissorving in oil of radioactive steel (irradiated with neutrons to Fe⁵⁹) or copper (activated by introducing tracer amounts of Ag¹¹⁰ into the molten copper) under the action of the additive being tested [1, 2, 16].

we studied the chemical activity with respect to radioactive copper and steel of various organic compounds of sulfur and chlorine which have found application as anti-abrasive additives. As an example, we have listed in Fig. 1 the results of a recommetric determination of the chemical activity of chlorinated paraffin, the sulfur-containing additive L2-0/9, and their mixtures, as well as mixture of dibenzydisulfide and chlorinated paraffin all of which had been

added to DC-14 oil. This determination was made with a copper plate at a temperature of 150°. In Fig. 1 we see that the chemical activity of the sulfur-containing additive and the chlorinated paraffin is considerably less than the chemical activity of their mixtures.

It is also seen that the mixture of dibenzydisulfide and chlorinated paraffin possesses a very high chemical activity, a fact which is also noted in the other papers [12]. According to data from tests on a four-ball apparatus this mixture provides the highest anti-abrasive effect.

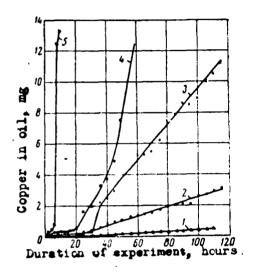


Fig. 1. Corrosion of a radioactive copper plate under the action of oil LC-14 with various auti-abrasive edditives. Pinkevich apparatus, oil temperature 150°.

- 1) Gil DC-14; 2) DC-14 oil with 7% entorinated paraffin; 3) DC-14 with 5% LZ-6/9;
- 4) DG-14 with to Lu-0/9 and % concentrated paraffin; 5) DG-14 with % dibenzyl-disulfide and 7% chlorinated paraffin.

Various additives were introduced into the cil along with the dibenzyldisulfide and chicrinated paraffin for the selection of the anti-corrosion
component. Several of them provided a decrease in corresion with copper and
steel. In Table 1 are listed the results of the relignative determinations and
the date from the tests on the four-bell machine for an oil of selective

refinement (with a viscosity, 100 = 14 cs) with the most effective of the anti-corrosion additives tested, 1-L. As seen in Table 1, the addition of

TABLE

	Corresion,		on four-	
Sample .	copper at 150° for 10 bours	ateal at 200° for 75 bours	Results of tests on ball meebine, maxim stress before jummin	
Oil of selective re- finement with viscos- ity, $\nu_{100} = 14$ es	0,03	-	79	
The same + 3% dibensyl disulfide + 7% chlorin ated paraffin		0,18	126	
The same + 3% dibenzyl disulfide + 7% shlorin paraffin + 6% additive *1-L.*	ated			
	0,8	0,042	126	

additive 1-L sharply decreases the corresive action of the oil with additives without impairing the anti-abrasive properties. However, the additive provides effective anti-corresive action with copper under the conditions of the test only for a period of 10 nrs.

Along with the empirical selection of anti-corrosion components, we also conducted a radiometric study of the anti-corrosive properties of phospherus-containing compounds, proceeding on the assumption that effective protection of the metal surfaces from corresion by enlorine and sulfur atoms can be attained via the creation of, not a molecular, but a more concrete stomic film through which it is more difficult for the mobile chronine and sulfur atoms to penetrate to reach the surface. It was assumed that such an atomic film might be obtained using phosphorus compounds. This assumption was based on data obtained by us

[•] Barium slaylphenoxide in a strution of hydrox, propylated akkyaphenol.

previously concerning the fact that the phosphides of the metals, in contrast to the sulfides, were insoluble products of the exidation of the oils, and are, therefore, held firmly on the surface of the metals for a long time [1-4]. Our attention last time was devoted to this as well as to other research [12]. For this, it is necessary that the phosphorus-containing compounds be soluble in the appropriate oil and that they eliminate phosphorus at a temperature considerably lower than that of decomposition of the anti-abrasive components. It was assumed, in addition to that, that the formation

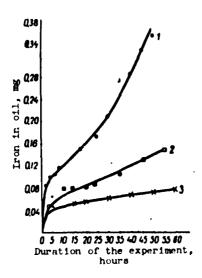


Fig. 2. Corrosion of radioactive steelplates under the action of an oil containing anti-abrasive additives and various anti-corrosion phosphorus-containing compounds. Pinkevich apparatus, oil temperature 200°. 1) oil of selective refinement with viscosity $v_{100} = 14$ cs + 3% dibenzyl disulfide + 7% chlorinated paraffin; 2) the same + 3% phosphorus-containing additive DF-9; 3) the same + 0.5% triphenyl phosphite.

on the surface of the metal of a phosphide, having an anti-wear effect, would not impair the effectiveness of the anti-abrasive components.

As is seen in Fig. 2, the addition of phosphorus additives assures a significant reduction in the corrosion of steel (at an oil temperature of 200°) under the action of a mixture of dibenzyl disulfide and chlorinated pareffin. In this way, according to the data from tests on the four-ball machine, the anti-abrasive effectiveness is not impaired. Listed below are the results of radiometric determinations of the corrosion of steel at an oil temperature of 200°.

	in 6 hrs, counts/min
011 DC-14	32
DC-14 + 3% L4-6/9 + 7% chlorinated paraffin	1648
The same + 0.5% tricresyl phosphate	1832
The same + 0.5% triphenvl phosphite	80

Corrosion of steel

The difference in temperature of decomposition(elimination of phosphorous) of triphenyl phosphite and tricresyl phosphate was evidenced by the effectiveness of the anti-corrosive action of these compounds in composition with a chemically active mixture of anti-abrasive components.

Tricresyl phosphate gave no anti-corrosive effect, while triphenyl phosphite provided a sharp decrease in corrosion. The proposed application of phosphorus-containing anti-corrosive components in composition with the most chemically active anti-abrasive additives can enable us to use as highly effective additives chemical compounds which have previously been rejected due to their high corrosive activities. The chemical interaction of the surfaces of friction with anti-abrasive additives, which prevent and lessen abrasion and seizing under conditions of high stress and contact temperatures, can in the presence of small and moderate stresses give rise to chemical wear, in some cases considerably exceeding that produced during operation on oils without additives.

Tests of an oil with anti-abrasive additives were conducted on a machine the friction-producing unit of which (Fig. 3) consisted of two hollow, cast-iron cylinders, with rubbing ends (lo mm in external diam.), one of which was radioactive [17]. The tests were conducted at 600 rpm with a force of 2.5 kg/cm² for a duration of 1 hours. The experiment was repeated many times and for an indication of the wear the average value of the wear from the data of all the parallel experiments was taken.

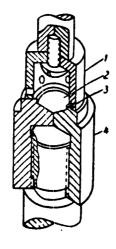


Fig. 3. Diagram of the frictionproducing unit of the machine. 1) radioactive sample subjected to to friction; 2) reservoir for the oil; 3) insert of cobalt-60; 4) non-radioactive sample subjected to friction.

From the list of results we see that the addition of anti-abrasive additives to DC-14 sharply increased wear on the friction surfaces. The use of these additives in composition with triphenyl phosphite (done to eliminate corrosion as

previously shown) insures that the level of wear on the surfaces of friction will be equal to the level of the wear which takes place when operating on oil without additives.

Thus, it has been experimentally proven that the use of phosphorus compounds as anti-corrosive components in composition with anti-abrasive additives not only sharpl, reduces corrosion, but also provides a decrease in chemical wear on the surfaces of friction with low stresses.

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